
2-अमीनो-4-नाइट्रोफेनॉल, तकनीकी — विशिष्टि

(पहला पुनरीक्षण)

2-Amino-4-Nitrophenol, Technical — Specification

(First Revision)

ICS 71.080.90

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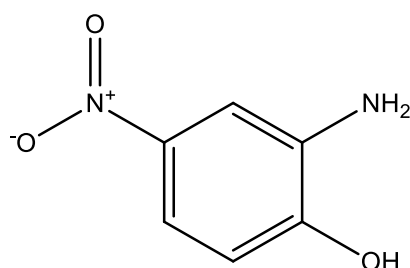
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FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

2-amino-4-nitrophenol ($C_6H_6O_3N_2$), is an important dye intermediate used in the manufacturing of azo dyes. It has the following structural formula:



2-AMINO-4-NITROPHENOL
MOLECULAR MASS: 154
CAS NUMBER: 99-57-0

2-amino-4-nitrophenol is harmful to aquatic life with long lasting consequences. Therefore, release of 2-amino-4-nitrophenol in environment should be prevented. Further, contents/containers are disposed of in accordance with national regulations as the material is suspected of causing cancer.

This standard was first published in 1989. The committee responsible for the preparation of this standard decided to revise it in order to incorporate recent development in analytical techniques and use of more sophisticated instrument to determine purity and impurity profile.

In this revision, test methods for determination of assay by perchloric acid and HPLC have been incorporated. Characteristic of colour of diazotized filtered solution, 2-aminophenol and 2,4-dinitrophenol have been deleted from the specification. Description of the material is also modified.

The bags in which the material is stored or transported may also be labelled with pictograms, signal word, hazard statement, and precautionary statement as given in Annex C, which are derived from GHS guidelines. At the time of publication, the latest edition of GHS guidelines was referred and are subject to revision and parties to agreement, are encouraged to investigate the possibility of applying the most recent labels as indicated.

The composition of the Committee responsible for formulation of this standard is given in Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Indian Standard***2-AMINO-4-NITROPHENOL, TECHNICAL — SPECIFICATION***(First Revision)***1 SCOPE**

This standard prescribes the requirements, the methods of sampling and testing for 2-amino-4-nitrophenol, technical.

2 REFERENCES

The standards given below contain provisions, which through reference in the text constitute provisions of this standard, at the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreement, based on the standard are encouraged to investigate the possibility of applying the most recent edition of these standards:

<i>IS No.</i>	<i>Title</i>
IS 1070 : 2023	Reagent grade water — Specification (<i>fourth revision</i>)
IS 5299 : 2001	Methods of sampling and tests for dye intermediates (<i>first revision</i>)
IS 14887 : 2014	Textiles — High density polyethylene (HDPE)/polypropylene (PP) woven sacks for packing 50 kg food grain — Specification (<i>first revision</i>)

3 REQUIREMENTS**3.1 Description**

The material shall be in the form of brownish yellow powder and shall be free from visible impurities.

3.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in col (4) and col (5) of Table 1.

4 PACKING AND MARKING**4.1 Packing**

The material shall be packed in HDPE/PP woven sacks (see IS 14887). Each package shall be securely closed.

4.2 Marking

4.2.1 Each bag shall bear legibly and indelibly the following information:

- Name of the product;
- Name of the manufacturer/supplier, complete address and recognized trade-mark, if any;
- Gross, net and tare mass;
- Lot or batch number.
- Month and year of manufacturing;
- Shelf life of the material; and
- Any other statutory requirement.

4.2.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

5.1 The method of drawing representative samples of the material shall be as prescribed in **4** of IS 5299.

5.2 Number of Tests

5.2.1 Test for assay by nitrite value and per chloric titration shall be conducted on each of the individual samples.

5.2.2 Tests for determination of all other characteristics, namely assay by HPLC, matter insoluble in dilute hydrochloric acid solution, given under Table 1, shall be conducted on the composite sample.

5.3 Criteria for Conformity**5.3.1 For Individual Samples**

The lot shall be declared as conforming to the requirement of assay by nitrite value and per chloric titration, if each of the individual test results satisfies the relevant requirements given in Table 1.

5.3.2 For Composite Samples

For declaring the conformity of a lot to the requirements of all other characteristics tested on the composite sample, the test results for each of characteristics shall satisfy the relevant requirements in Table 1.

6 TESTS

6.1 Preparation of Sample

Dry the material at $(105 \pm 1)^\circ\text{C}$ to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the

sample to an atmosphere containing acidic or alkaline fumes. Use this prepared sample for tests.

6.2 Tests shall be carried out on the prepared sample (see 6.1) according to the methods prescribed in col (4) and col (5) of Table 1.

6.3 Quality of Reagents

Unless specified otherwise, 'pure chemicals' and distilled water (see IS 1070) shall be employed in tests.

NOTE — 'Pure Chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirement for 2-Amino-4-Nitrophenol

(Clauses 3.2, 5.2.2, 5.3.1, 5.3.2 and 6.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	Clause No. of IS 5299
(1)	(2)	(3)	(4)	(5)
i)	Assay by			
	a) Nitrite value, percent by mass (on dry basis), <i>Min</i> or	75.0	A-2	—
	b) Per chloric titration, percent by mass (on dry basis), <i>Min</i> or	70.0	A-3	—
	c) Purity (by HPLC ¹⁾), percent area (on dry basis), <i>Min</i>	99.0	B	—
ii)	Matter insoluble in dilute hydrochloric acid solution, percent by mass, <i>Max</i>	0.20	—	11.1

¹⁾ In case of disputes, assay by HPLC shall be the referee method.

ANNEX A

[Table 1, Sl No. (i)]

DETERMINATION OF 2-AMINO-4-NITROPHENOL CONTENT (ASSAY)

A-1 GENERAL

Three methods namely, Method A determination of assay by nitrite value; Method B determination of assay by perchloric titration and determination of assay by HPLC (Annex B) have been prescribed. In case of disputes, determination of assay by HPLC (Annex B), shall be the referee method.

A-2 METHOD A

A-2.1 Reagents

A-2.1.1 Concentrated Hydrochloric Acid

A-2.1.2 Potassium Bromide

A-2.1.3 Standard Sodium Nitrite Solution — 0.1 N

A-2.1.4 Potassium Starch Iodide Indicator Paper

A-2.1.5 Ice

A-2.2 PROCEDURE

Weight 10 g to 14 g dry powder in 250 ml glass beaker. Add distilled water approximately 150 ml and stir with glass rod to make a smooth slurry. Add 20 percent soda ash solution (approximately 7 ml to 10 ml) to dissolve the powder to make clear solution. Transfer the solution to 500 ml volumetric flask along with little distilled water wash. Make volume exactly 500 ml by adding distilled water. Stir the contents well with magnetic stirrer. Take 50 ml of the solution by using pipette in to 1 000 ml beaker. Add 200 ml to 250 ml distilled water. Add ice cubes to make the temperature around 10 °C. Weigh and add 1 g Potassium bromide into the cold solution. Add Hydrochloric acid to make the pH acidic (pH around 2 to 2.5 on pH paper) approximately 25 ml is required. Take 0.1 N sodium nitrite in the burette. Titrate this solution against 0.1 N sodium nitrite solution with constant stirring by using magnetic stirrer. Check the end point to put the spot-on starch iodide paper, the end point shows faint blue ring on starch iodide paper. Check the sodium nitrite solution consumed by burette reading.

A-2.3 Calculation

Assay (by nitrite value), percent by mass (on dry basis) =

$$\frac{V \times N \times 154}{M}$$

where

V = volume, in ml, of standard sodium nitrite solution used in the titration;

N = normality, N, of sodium nitrite solution; and

M = mass, in g, of the dry material taken for the test.

A-3 METHOD B

A-3.1 Reagents

A-3.1.1 Per-Chloric Acid — 0.1 N

A-3.1.2 Alpha Naphthyl Benzene

A-3.1.3 Acetic Anhydride

A-3.1.4 Acetic Acid Glacial — AR grade

A-3.2 Procedure

Weigh accurately between 0.20 g to 0.40 g of sample in 250 ml dry conical flask. Dissolve the sample in 50 ml of acetic acid. Add 5 ml of acetic anhydride solution. Add alpha naphthyl benzene as an indicator and titrate with the 0.1 N per chloric acid till light yellow to green colour change considered end point.

A-3.3 Calculation

Assay [by perchloric acid (HClO₄) titration], percent by mass (on dry basis) =

$$\frac{V \times N \times 154 \times 100}{1\ 000 \times M}$$

where

V = volume, in ml, of per chloric acid solution used in the titration;

N = normality, N, of per chloric solution; and

M = mass, in g, of the dry material taken for the test.

ANNEX B

[Table 1, Sl No.(i)]

DETERMINATION OF 2-AMINO-4-NITROPHENOL CONTENT (ASSAY) BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

B-1 OUTLINE OF METHOD

High performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry so as to identify, quantify or purify the individual components of the mixture.

B-2 APPARATUS

B-2.1 Isocratic Liquid Chromatography System — with UV detector capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

B-2.1.1 Column — C18 column of 100 Å with length 250 mm, internal diameter 4.6 mm and particle size 5 µm or equivalent.

B-2.2 Analytical balance — having least count of .001 g.

B-3 REAGENT

B-3.1 Tetrabutylammonium Hydrogen Sulphate — AR grade

B-3.2 Dipotassium Hydrogen Phosphate — HPLC grade

B-3.3 Methanol — HPLC grade

B-3.4 Water — HPLC grade

B-3.5 Acetonitrile — HPLC grade

B-3.6 2-Amino-4-Nitrophenol — pure substance of known purity

B-4 SAMPLE PREPARATION

Weigh accurately 0.010 g (10 mg) of dry 2-amino-4-nitrophenol in 100 ml volumetric flask.

Dissolve it in water and make up to 100 ml with water.

B-5 BUFFER PREPARATION

Take 12 g Tetrabutylammonium hydrogen sulphate (TBAHS) and 29 g Dipotassium hydrogen phosphate (K_2HPO_4) in 190 ml water and dissolve (stock solution).

B-6 FLOW RATE — 1ml/min

B-7 MOBILE PHASE — 96 ml water + 4 ml Buffer + 70 ml Acetonitrile + 7 ml Methanol

B-8 COLUMN OVEN TEMPERATURE — 26 °C

B-9 INJECTION VOLUME — 20 µl

B-10 RUN TIME — 10 minute, *Max*

B-11 WAVELENGTH — 254 nm

B-12 PEAK TIME

2-amino-4-nitrophenol : 4.0 min

B-13 CALCULATION

Calculate the peak area of individual constituent pertaining to 2-amino-4-nitrophenol the chromatogram of the material. The concentration of the constituent may be obtained on the basis peak area on chromatogram obtained with known amount of pure 2-amino-4-nitrophenol.

$$\text{2-amino-4-nitrophenol, percent} = \frac{A}{\text{Total Area}} \times 100$$

where

A = area of peak 2-amino-4-nitrophenol in sample.

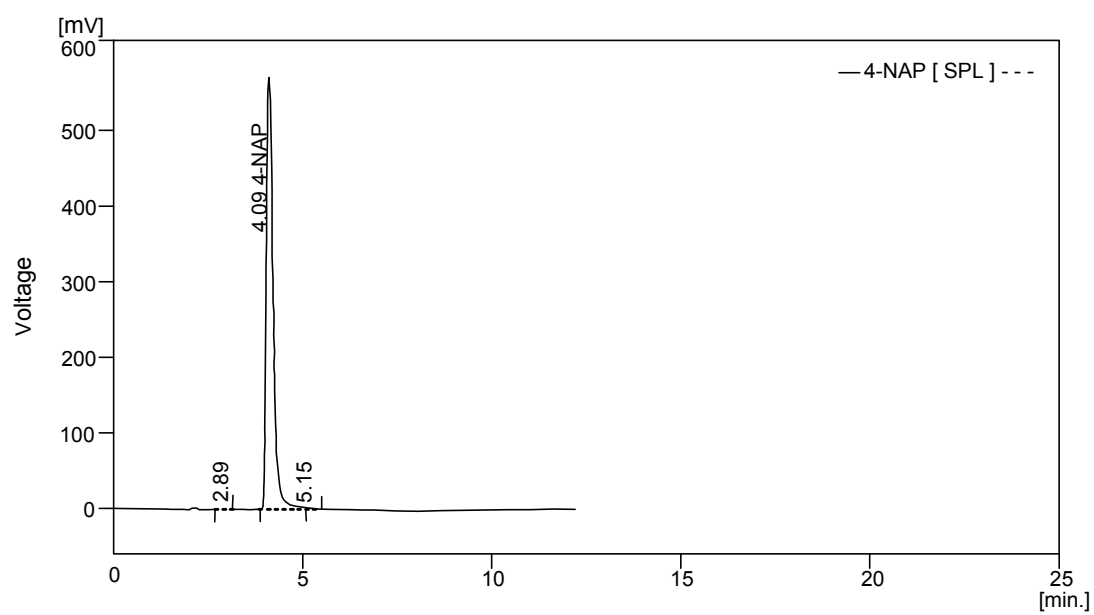


FIG. 1 TYPICAL CHROMATOGRAM

ANNEX C

(Foreword)

PICTOGRAMS, SIGNAL WORD, HAZARD STATEMENT AND PRECAUTIONARY STATEMENT

Pictogram(s)	:	
Signal Word	:	WARNING
Hazard Statement(s)	:	H317 : May cause an allergic skin reaction. H351 : Suspected of causing cancer. H410 : Very toxic to aquatic life with long lasting effects.
Precautionary Statement(s)	:	Prevention: P261 : Avoid breathing dust/fume/gas/mist/vapours/spray. P272 : Contaminated work clothing should not be allowed out of the workplace. P273 : Avoid release to the environment P280 : Wear protective gloves/protective clothing/eye protection/face protection Response: P363 : Wash contaminated clothing before reuse P391 : Collect spillage. Hazardous to the aquatic environment P302 + P352 : If on Skin : wash with plenty of soap and water P308 + P313 : If exposed or concerned: get medical advice/attention Disposal: P501 : Dispose of contents/container in accordance with local/regional/national/International regulations. Waste treatment in accordance with national regulations.

ANNEX D*(Foreword)***COMMITTEE COMPOSITION**

Dye Intermediates Sectional Committee, PCD 26

<i>Organization</i>	<i>Representative(s)</i>
Institute of Chemical Technology, Mumbai	PROF GANAPATI SUBRAY SHANKARLING (Chairperson)
Aarti Industries Limited, Mumbai	DR VAISHALI BHANDARY DR SANJEEV KUMAR DIXIT (<i>Alternate</i>)
Ankleshwar Research and Analytical Infrastructure Limited, Ankleshwar	SHRI MANSUKH H. VEKARIA
Alkyl Amines Chemicals Limited, Mumbai	SHRI S.V. NIKUMBHE SHRI KIRAT PATEL (<i>Alternate</i>)
Archroma India Private Limited, Thane	DR RAJESH RAMAMURTHY SHRI HEMANT MHADESHWAR (<i>Alternate</i>)
Atul Limited, Valsad	DR M. U. RAHMAN DR J. G. DESAI (<i>Alternate</i>)
BASF India Limited, Mumbai	SHRI UDAY KULKARNI
Central Revenue Control Laboratory, New Delhi	SHRI V. SURESH SHRI SHIVRAJ SINGH (<i>Alternate I</i>) SHRI MRITUNJOY MAITY (<i>Alternate II</i>)
Colourtex Industries Limited, Mumbai	DR PANKAJ DESAI SHRI R. K. JAISWAL (<i>Alternate</i>)
Deepak Nitrite Limited, Vadodara	SHRI SAILASH RAVAL SHRI RAJENDRA SHINDE (<i>Alternate</i>)
Defence Research Development Organization, Ministry of Defence, New Delhi	DR PRADEEP K. GUPTA
Dystar, Mumbai	DR MONIKA SINGH
Ecological and Toxicological Association of Dyes, Vadodara	DR PARITI SIVA RAMA KUMAR
Gujarat Dyestuffs Manufacturers Association, Ahmedabad	SHRI YOGESH PARIKH SHRI ANIL M. JAIN (<i>Alternate I</i>) SHRI HARESH BHUTA (<i>Alternate II</i>)
Gujarat Narmada Valley Fertilizers Company Limited, Ahmedabad	SHRI R. M. PATEL SHRI C. S. PATEL (<i>Alternate</i>)
Gujarat Pollution Control Board, Gandhinagar, Ahmedabad	SHRI D. M. THAKER
Heubach Colour Private Limited, Mumbai	SHRI J. I. SEVAK

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<i>Organization</i>	<i>Representative(s)</i>
Huntsman Textile Effects, Mumbai	MS PRACHI JOSHI
Indian Beauty and Hygiene Association, Mumbai	MS MALATHI NARAYANAN
Indian Chemical Council, Mumbai	SHRI P. S. SINGH
Jay Chemicals Industries Private Limited, Ahmedabad	SHRI VILPESH YADAV SHRIMATI MAITRI VYAS (<i>Alternate</i>)
Kiri Industries Limited, Ahmedabad	DR GIRISH H. TANDEL SHRI YAGNESH MANKADR (<i>Alternate</i>)
Meghmani Dyes and Intermediates Limited, Ahmedabad	SHRI MANOHAR MAHESHWARI SHRI RAMESH SHINGARE (<i>Alternate</i>)
Ministry of Environment Forest and Climate Change, New Delhi	SHRI N. SUBRAHMANYAM
Sudarshan Chemical Industries Limited, Pune	DR R. SRIDHARAN
The Bombay Textile Research Association, Mumbai	MS SHITAL PALASKAR
The Dyestuff Manufactures Association of India Office, Mumbai	SHRI V. R. KANETKAR
BIS Directorate General	SHRIMATI MEENAL PASSI, SCIENTIST 'F'/ SENIOR DIRECTOR AND HEAD (PETROLEUM, COAL AND RELATED PRODUCTS) [REPRESENTING DIRECTOR GENERAL (<i>Ex-officio</i>)]

Member Secretary
SHRI ABHIJEET SINGH
SCIENTIST 'B'/ASSISTANT DIRECTOR
(PETROLEUM, COAL AND RELATED PRODUCTS), BIS

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Amend No.	Date of Issue	Text Affected

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